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Canola meal moisture-resistant fuel pellets: Study on the effects of process variables and additives on the pellet quality and compression characteristics

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ABSTRACT

This study focuses on pelletization of waste canola meal biomass to increase the bulk density, thereby reducing the transportation and storage costs, thus provide better material feeding in gasification reactors with less dust formation. The effects of feed constituents of canola meal such as protein, fiber, fat, lignin and feed moisture content as well as added binder, lubricant and densification process parameters on the strength and durability of the densified product are investigated. The increased durability (99%) of canola meal pellets was a result of added binder (5 wt%) and the inherent protein (40 wt%) and lignin (12 wt%) content in the feed. From the compression data at different temperature and pressure, Kawakita and Lüdde model (1971) was developed to classify the feed material into groups. The R^2 value $\geq$0.999 showed good model fit. It was found that at temperature >70 °C, the particle undergoes rearrangement followed by fragmentation and particle plastic deformation during the compression process. The effects of coating agent on pellet durability, hardness and moisture uptake were studied to produce moisture-resistant pellets. Finally, the pellets were gasified in a fixed bed reactor using different gasifying agents such as steam, oxygen (O_2) and carbon dioxide (CO_2) and their effects were assessed. Carbon dioxide was found to give maximum carbon efficiency (CE) up to 82.7% and 50.7 MJ/m^3 LHV of gas at a temperature of 750 °C and equivalence ratio (ER) of 0.4., whereas O_2 gave 66.5% of CE with 44.7 MJ/m^3 LHV of gas at 650 °C and 0.4 ER and steam produced gas with LHV 40.8 MJ/m^3 with CE 27.4% at 650 °C and 0.2 ER. Thus, by producing moisture-resistant canola meal pellets with reasonable fuel characteristics, pelletization of canola meal provides a promising alternative for the utilization of canola meal waste as an alternative source of renewable energy.

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1. Introduction

Biomass residues are a valuable solution for generating power and can offset greenhouse gas emission, without being dependant on seasonal and weather changes which affect solar and wind power. Biomass can be stored and continuously utilized for heat, energy or power production (Wolfgang et al., 2012). Considering the energy resources such as fossil fuel, coal and oil in the world, agricultural residues or wastes were found to be as third largest energy resource (Bapat et al., 1997). The agricultural residues (wood, agricultural stovers, grasses and lignocellulosic material) are renewable and sustainable, and can help in reducing the carbon emission as compared to fossil fuel (Tumuluru et al., 2010).

The low bulk density of biomass (<150 kg/m^3), irregular shapes and different sizes limit their transportation, storage and utilization in actual form (Gilbert et al., 2009; Bowyer and Stockmann, 2001; Sokhansanj et al., 2006). To overcome these limitations, there is a need to develop more efficient methods for densification of biomass. The densification of biomass increases the density of biomass pellets typically >600 kg/m^3 and helps to reduce the transportation costs with convenient material handling and less dust formation (Gilbert et al., 2009). Densification of biomass into pellets, briquettes or cubes makes the material in uniform shape and sizes for easy handling, which can be directly used

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for thermochemical processes such as combustion, gasification, pyrolysis or co-firing with coal (Kaliyan and Morey, 2009). Increase in crude oil and gas prices led to rapid development in biomass pellet industries with more emphasis to reduce greenhouse gas emission (Peng et al., 2013). Commercial densification of biomass is typically carried out by conventional pressure driven processes such as extrusion, pelletization and briquetting (Li and Liu, 2000; Kaliyan and Morey, 2009). To prepare the densified quality biomass product, it is essential to study desirable and dependent parameters such as density and durability in relation to independent parameters such as moisture content (Adapa et al., 2007).

Pellets are more vulnerable to physical wear and tear during transport and storage. This leads to the formation of fine particles or dust which can create problems in the boiler or combustion systems (Carroll and Finnan, 2012). In addition, it can be a source of both health and fire hazard. As per the European Committee for Standardization (CEN), CEN/TS 15210 method defines the physical durability as the ability of pellet to remain undamaged during transportation. It means the ability of pellet to survive vibrations and shock. During the densification process, to a great extend the physical (thermal, mechanical or atomic) forces determine the strength of pellet produced (Adapa et al., 2002). Therefore, it is essential to understand the fundamental mechanism behind the biomass compression process, and to design energy efficient combustion process (Mani et al., 2004).

Continuous increase in demand for biomass pellets and the inadequate availability of agricultural resources has resulted in finding out new available raw materials for pellet production from various resources such as fibrous residue, straw, husks, stover, pulps, meal, grass and wastes from the food industry. The recent Canadian government directive to substitute 1.5 billion liters of petroleum based diesel per year (5%) with biodiesel by the year 2018 are projected to encourage biodiesel industries (Canola Council, 2012). The immense development of biodiesel industries in upcoming years will produce by-products in large quantity such as oilseed meal, for which their utilization will become uncertain. Particularly in Canada, biodiesel industries utilize canola as a common feedstock for the biodiesel production, resulting in abundant quantities of waste canola meal. Apart from being more widely used as an animal feed, canola meal can be used as alternative feedstock for biofuel/bioenergy production (Tilay et al., 2014; Azargohar et al., 2013). During 2013, Canada has 41 pellet manufacturing plants with total capacity of >3 million tons of annual production and has exported ~94% to overseas (Natural Resources Canada, 2013).

Biomass pellets offer ecological advantages over traditional fuels such as heating oil and natural burning gas. Biomass pellets in a pellet stove produce smaller volumes of hydrocarbons (methane) and CO_2. In general, pellet mills need a cheap and reliable source of waste materials due to rising competition for biomass. The competition and prices for renewable feedstock fluctuate as emerging applications such as alternative biomass, advanced biofuel, biochar from biomass, activated carbon made from the petcoke compete for these resources. Our previous study investigated that the waste canola meal can be successfully utilized as an alternative source for renewable energy (Tilay et al., 2014). The overall goal of this study is to develop quality fuel pellets from waste canola meal and to provide optimized operating conditions to produce more durable fuel pellets. The study was carried out as follows: (1) to study the effects of process variables (compressive force, temperature) and feedstock variables (moisture content and binder) on the quality of canola meal pellet; (2) to study the change in density and volume of pellets as a function of pressure followed by development of compression model; (3) to study effects of coating agent on the moisture-resistant property of pellets and (4) to study the effects of different gasifying agents on the quality of syngas produced using pellet gasification in a fixed bed reactor.

2. Materials and methods

2.1. Materials

The waste canola meal biomass from Milligan Biofuels Inc. (Saskatchewan, Canada) were used as raw materials for the present study. The proximate analysis of biomass was previously carried out using AAFCO (Association of American Feed Control Officials) standard (Tilay et al., 2014). The canola meal material was ground by means of knife mill (Retsch GmbH, 5657 HAAN, West Germany) and passed through 0.8 mm mesh. Further, the particle size distribution of the ground canola meal was determined using Mastersizer 9000 laser-scanning particle size analyzer (Malvern Instruments Ltd., Malvern, UK) which confirms that 80.3% particles were in the range of 100–800 μm. The volume of fine (<100 μm) and coarse (>1000 μm) particles was determined to be 14.2 and 5.5%, respectively. The moisture content of the ground canola meal was determined using ASTM 3173-87 method and was 5.19 ± 0.8% as received. Similarly, ash content of the manufactured pellet was determined as per ASTM 3174-04 in a laboratory muffle furnace (Holpack, USA) and was in the range of 5.5–5.7 wt%, depending upon the composition of feed and additives. The additives (binder, lubricant and coating agent) used in this study were procured from Evergreen BioFuels Inc. (Montreal, QC, Canada). All addetives used were provided by Evergreen BioFuels Inc. and their resources and chemical compositions were not disclosed. The elemental analysis of procured binder and pellet for carbon (C), hydrogen (H), nitrogen (N), and sulfur (S) was performed using a Perkin–Elmer Elementar CHNSO analyzer (Vario EL III, Elementar Americas Inc., NJ) and the analyzer calibration was done using standard sulfanilic acid. The elemental analysis of binder showed 2.3 ± 0.05 wt% of N; 49.0 ± 0.2 wt% of C; 0.2 ± 0.07 wt% of S and 5.3 ± 0.07 wt% of H. In case of canola meal pellet using optimized formulation, the composition elements were found to be around 6.1 ± 0.07 wt% of N; 46.6 ± 0.8 wt% of C; 0.9 ± 0.3 wt% of S and 6.6 ± 0.2 wt% of H. The higher heating value (HHV) of produced canola meal pellet was measured by oxygen bomb calorimeter (Parr® 6400 Calorimeter, IL, United States) using ASTM D5865. The canola meal pellet sample was burnt in a Parr 1108, placed inside a Parr 1341 isothermal calorimeter. Approximately 1 mL of water was added to the bomb and pressurized to 2.5 MPa, before placing in an isothermal jacket filled with 2000 ml of water at room temperature (25 ± 1 °C). The electrical energy (40 V) was applied for ignition using a platinum wire. The test was carried in triplicates. All the produced canola meal pellets were found to have HHV of ~20.3 ± 0.18 MJ/kg.

2.2. Preparation of sample and densification

The desired quantity of moisture was added to the formulation to make the final moisture content in the range of 8–12 wt%. The additives (binder and lubricant) were added to the pre-adjusted moisture of canola meal, in the range of 2–5 wt% and 1–3 wt%, respectively and kept in air tight seal bags for 12 h. All samples were densified in a lab scale single-pelleting unit used in previous studies as described by Adapa et al. (2013) and Kashaninejad and Tabil (2011). The densification unit was composed of a plunger-die assembly. The internal diameter and length of a steel cylinder assembled on the Instron testing machine (3360 Dual Column Tabletop Testing Systems, Instron Corp., Norwood, MA) were 6.5 mm and 135.3 mm, respectively, and fitted with a 10,000 N load cell. The die was surrounded with a dual element heating tape (Cole-Parmer Instrument Company, Vernon Hills, IL) to maintain the desired temperature during the densification process. One thermocouple (type-T) was connected to the outer surface of the die and another to a temperature controller. The die was positioned on a raised base which consist of sliding gate at the bottom allowing the

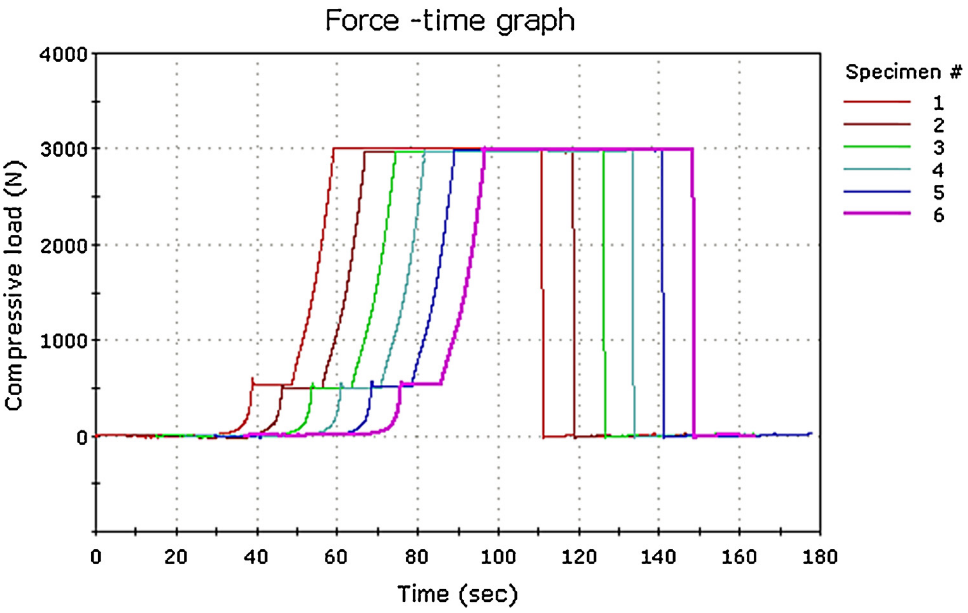

Fig. 1. Force–time graph (densification method).

ejection of pellet after the densification process. The plunger was attached to the upper moving crosshead of the testing machine.

Once die reached a constant set temperature of 60 ± 1 °C, a weighed quantity of sample (0.8 ± 0.02 g) was loaded into the die. During the densification process, initially compressive force of 500 N (equivalent pressure 15.8 MPa) was applied to the sample. The plunger moves down to the pre-set speed 50 mm/min and stops for 15 s at the applied load of 500 N (Fig. 1). This allows material to reach the desired set temperature. After 15 s, the plunger moves down with the same set speed and the pre-set final compressive force of 3000 N (equivalent pressure 94.7 MPa) was applied to densify the samples. Once the pre-set load was attained, the plunger stops and retains in place for 60 s for the relaxation test (Kashaninejad and Tabil, 2011) and in addition prevents spring back action of compressed sample (Mani et al., 2006). The plunger was withdrawn to release the applied load and the sliding gate was opened. After 30 s, the plunger moves down to eject the pellet. Each sample was determined in fifteen replicates. Following ejection, once the pellet sample attains ambient temperature, the mass, length, and diameter of pellet were measured using a digital caliper.

2.3. *Experimental plan*

2.3.1. *Box–Behnken design to study the effect of additives and moisture content on pellet quality*

Box–Behnken design (BBD) matrix was used to examine the influence of most important chemical parameters such as concentration of additives and moisture content. The three parameters: binder (A), lubricant (B) and moisture content (C) were considered to find the most suitable combination of these variables resulting high quality pellets. Different formulations were prepared (Table 1) using BBD (Stat-Ease, Inc., version 6.0.8) at three levels, coded as −1, 0, and +1. BBD creates designs with desirable statistical properties with only a fraction of the experiments required for a three-level factorial with the appropriate quadratic model. The design comprised three factors with three levels together with five replicates at the center point. These experiments were used to assess the linear and interaction effects of these factors considered and to fit a second order quadratic model. These factors were considered as an independent variable and pellet quality parameters (durability and hardness) were dependent variable. Lower, middle and high level of each variable were coded as −1, 0 and +1 respectively. The

Table 1
Effects of binder, lubricant and moisture content on pellet density, pellet relaxed density, durability and hardness of canola meal pellets made in the single pelleting unit.

Formulation	Binder (A) (%w/w)	Lubricant (B) (%w/w)	Moisture (C) (%db)	Pellet density (kg/m^3)	Relaxed density (kg/m^3)	Durability (%)	Hardness (N)
1	2	1	10	1160 ± 17	1165 ± 13	61 ± 7	23 ± 6
2	5	1	10	1183 ± 7	1177 ± 4	66 ± 11	48 ± 1
3	2	3	10	1161 ± 13	1170 ± 17	45 ± 7	41 ± 3
4	5	3	10	1161 ± 12	1181 ± 12	63 ± 4	47 ± 7
5	2	2	8	1172 ± 6	1155 ± 15	41 ± 10	30 ± 0
6	5	2	8	1172 ± 7	1144 ± 9	64 ± 5	25 ± 2
7	2	2	12	1173 ± 10	1193 ± 8	58 ± 13	30 ± 6
8	5	2	12	1181 ± 6	1190 ± 8	64 ± 13	71 ± 3
9	3.5	1	8	1167 ± 7	1151 ± 20	49 ± 6	21 ± 2
10	3.5	3	8	1160 ± 10	1145 ± 15	44 ± 10	52 ± 1
11	3.5	1	12	1185 ± 5	1214 ± 8	61 ± 13	63 ± 5
12	3.5	3	12	1168 ± 8	1195 ± 2	48 ± 15	35 ± 7
13	3.5	2	10	1163 ± 8	1168 ± 23	61 ± 9	40 ± 1
14	3.5	2	10	1162 ± 7	1167 ± 13	60 ± 10	40 ± 1
15	3.5	2	10	1163 ± 8	1168 ± 9	60 ± 8	39 ± 0
16	3.5	2	10	1163 ± 7	1169 ± 14	61 ± 7	41 ± 2
17	3.5	2	10	1165 ± 6	1169 ± 12	60 ± 8	41 ± 1

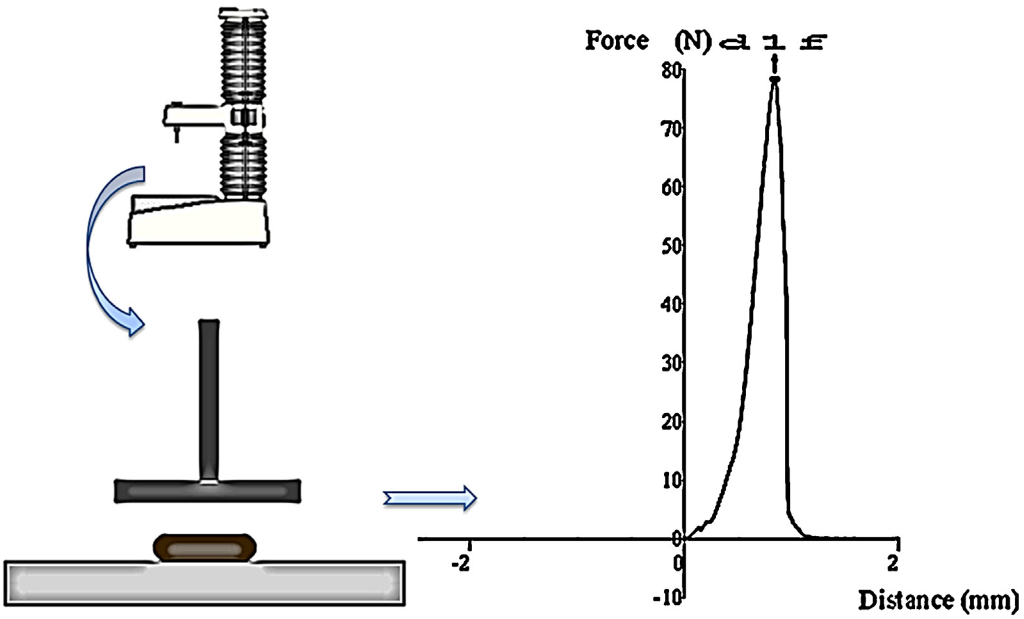

Fig. 2. Hardness testing of pellets using a texture analyzer and recorded force-distance curve to determine the force required to break the pellet.

actual values along with coded level are described in Table 1. A second order polynomial equation was adopted to find the effects of independent variables to the response. For predicting the best suitable combination, the following second-order polynomial equation was developed to correlate the relationship between selected independent variables and the dependent responses (durability and hardness):

$$\text{Response} = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_{11} A^2 + \beta_{22} B^2 + \beta_{33} C^2 + \beta_{12} AB + \beta_{13} AC + \beta_{23} BC \quad (1)$$

where the response is either durability or hardness of the pellet; β_0 – constant term; β_1, β_2 and β_3 – coefficient of linear terms; β_{11}, β_{22} and β_{33} – coefficient of quadratic terms; β_{12}, β_{13} and β_{23} – coefficient of cross product terms (two variables) respectively.

The quality of fit of the polynomial equation was expressed with the coefficient of determination R^2.

As per the BBD, each formulation was prepared by adding additives at different concentrations (please see formulations 1–17 in Table 1) and kept in an airtight sealed bags for 12 h at room temperature for even distribution of moisture before densification. Further, the densification process was carried out for all formulations at applied preset load of 3000 N and a temperature of 60 °C, using a lab-scale single pelleting unit as mentioned above. The optimized combination of formulation was selected on the basis of pellet durability and hardness. A sample without binder, lubricant and the desired moisture content was considered as control.

2.3.2. *Effect of physical parameters on pellet quality*

The optimized combination of pellet formulation obtained from BBD was used to study the effects of pressure and temperature on pellet quality. The preset loads used for these tests were 3500, 4000 and 4500 N and a temperature of 60 °C, at a crosshead speed of 50 mm/min. Further, the optimized load was considered depending on pellet durability and harness, to study the effects of temperature (70, 80 and 90 °C) at an optimized preset load. In each compression test, the sample (0.8 ± 0.02 g) was fed into the heated die and compressed up to the stated preset load and held for 60 s to arrest the spring back effect. Specified samples from each study were tested for density, durability and hardness measurement.

2.3.3. *Durability and pellet density measurement of a single pellet*

Durability is one of the key parameters used to describe the physical quality of the pellets. There are different methods investigated for durability test (Temmerman et al., 2006). The tumbling method for pellet durability test (ASABE 269.4) described by the American Society for Agricultural and Biological Engineering (ASABE) was mostly accepted with high accuracy level and limited test replicates required. Due to the limited quantity of pellets, drop test as described by Adapa et al. (2010) was performed to measure the durability of canola meal pellets obtained after pelletization. Pellet sample (ten replicates) was dropped from a height of 1.85 m on a metal plate. The ratio of mass retained with the initial weight was expressed as the percentage durability of the pellet (Adapa et al., 2010; Al-Widyan and Al-Jalil, 2001).

Following the extrusion of the pellets, the length, mass and diameter of the pellets were measured using a calibrated digital caliper to determine the density in kg/m^3. Five replicates (pellets) from each different experiments were considered. The process was repeated to find out the change in pellet density (% expansion/relaxed density) after a storage period of two weeks (Adapa et al., 2010; Serrano et al., 2011).

2.3.4. *Pellet hardness test*

The internal strength of the produced pellets was measured using a compression test by applying a load at a constant rate, until the test pellet breaks. The load at fracture is recorded as the hardness and reported as force (N). Hardness was related to the chewability or palatability of pellets previously (Adapa et al., 2006; Mahapatra et al., 2010). The hardness of canola meal pellets was measured using a TA-XT2i texture analyzer (Stable Micro Systems, Scarsdale, NY). A single pellet 20 ± 1 mm in length and 6.4 ± 0.05 mm in diameter was placed by positioning horizontally on a flat surface (Fig. 2) under the disk shaped metal probe (dia-35 mm) attached to 100 kg load cell. A test was carried out by increasing the applied load at a constant rate of 2 mm/s, until the pellet failed by cracking or breaking and stopped after pellet failure. The average force required to break the pellet was calculated based on five replicates per sample (Mahapatra et al., 2010). Post run, the load cell comes to its original position with the preset speed of 10 mm/s. The maximum force needed to break the pellet sample was determined directly by the software (Stable Microsystem version 2.64) and taken as hardness (Adapa et al., 2006).

2.3.5. Bulk and particle density

The bulk density (kg/m^3) of biomass was determined using a 250 ml graduated measuring cylinder filled using a funnel. The cylinder was tapped on a table for approximately 15 times to let the material to settle down and subsequently the container was weighed. The bulk density was determined in replicates of five. Similarly, the particle density (kg/m^3) of the ground canola meal was measured using a gas multi-pycnometer (Quanta Chrome, Boynton Beach, FL) by calculating the displaced volume of nitrogen gas with a known mass of sample (Adapa et al., 2010). The particle density measurements were performed in triplicates.

2.3.6. Compression model

This study represents a protocol for the assessment of mechanical properties of a ground canola meal, and evaluates the pelleting relevant information carried forward by compression data. This is useful in a pelletization process to enhance and understand the process and also applicable for monitoring of the pelletization process. Various compression models applied to pharmaceutical and biomass materials have been studied and reviewed earlier in detail (Adapa et al., 2002, 2009; Mani et al., 2003; Denny, 2002). For agricultural non-treated biomass such as barley, canola, oat, and wheat straw (1.98 mm grind size), Kawakita and Lüdde (1971) model was delivered as best fit in addition to deformation characteristics using Cooper and Eaton (1962) and Jones (1960) model by Adapa et al. (2009). Kawakita and Lüdde (1971) compression model was developed to explain the compaction process during the pelletization.

Kawakita and Lüdde model

One of the ways of representing compression data observed was to establish the relationship between applied pressure and reduction of a powder bed. The proposed equation for compaction of powders based on pressure and volume (Kawakita and Lüdde, 1971; Kawakita and Tsutsumi, 1965) (Eq. (2)).

$$\frac{P}{C} = \frac{1}{ab} + \frac{P}{a} \qquad (2)$$

where C is the degree of volume reduction: $C = (V_o - V)/V_o$, V_o is the initial volume of the powder bed, V is the volume under applied pressure, P is the applied pressure, and 'a' and 'b' are parameters.

The Kawakita equation includes two compression parameters referred to as 'a' and 'b'. The parameter 'a' characterizes the engineering strain or degree of compression at infinite pressure (C_∞), while the inverted b-parameter characterizes the applied pressure required to achieve an engineering strain of $C/2$ (Nordstrom et al., 2008). The importance of Kawakita parameters has been discussed in terms of the physical properties of the particles (fracture strength) and the yield pressure of the particle (Nordstrom et al., 2008). Thus, it is reasonable that the original size of the feed particles can affect the compression process, and consequently the values of the Kawakita parameters. It is hypothesized that the corresponding effects on the Kawakita parameters during compression of a fine powder will be a low value of parameter 'b^{-1}' and a high value of the parameter 'a' (Nordström et al., 2009). The combination of Kawakita parameters 'a' and 'b' reveals the occurrence of particle rearrangement during the compression process. This is relatively important depicting the initial stage for the overall compression behavior.

The value of parameters 'a' and 'b' can be derived from a linear relationship between P/C and P. Materials for which particle rearrangement has a substantial influence on the overall compression process, are related to the low value of parameter 'b^{-1}' and high value of parameter 'a'. The product of these Kawakita parameters is called as an index (ab_I) from which a material can be classified as either Class I ($ab_I > 0.1$) or Class II ($ab_I \leq 0.1$). A material showing a combination of $ab_I > 0.1$, $a > 0.6$ and $b^{-1} < 7$ is thus typical features for a Class I powder (Nordström et al., 2012).

The optimized formulation obtained from BBD was used for this study. The single pelleting unit was used to make pellets from canola meal. The sample loading for making pellets was kept constant at 0.80 ± 0.02 g. The experimental parameters (temperature and applied pressure) were studied at different ranges. The pellet die temperature was studied at different temperatures of 60, 70, 80 and 90 (±1 °C) in order to simulate the heating during the commercial pelleting process. Four preset pressures of 2500, 3000, 3500 and 4000 N corresponding to pressures of 75.37, 90.44, 105.52 and 120.59 MPa were used to compress samples. The crosshead speed was set to 50 mm/min. Once the preset load was attained, the plunger remains in place for 60 s in order to avoid spring-back effect of biomass (Adapa et al., 2006). Later, the pellet was ejected and kept for cooling at ambient temperature. The weight, length and diameter of pellet were measured.

2.3.7. Pellet coating and storage study

The final pellet formulation obtained from BBD was used to make pellets at an optimized temperature of 90 °C and applied load of 3500 N. These pellets were used for coating studies. Freshly prepared pellets were coated using the coating agent (4% dissolved in ~85% isopropyl alcohol). Further, pellets were heat cured in an oven at a temperature of 100 °C for 30 s. Obtained coated pellets were stored in an open atmosphere and at an ambient temperature with humidity ~60%. To study the effect of coating agent on moisture resistant canola meal fuel pellets, experiments in triplicates were performed at an interval of 1 week for each set of moisture content, durability and hardness study following up to 8 weeks. For comparison, control pellets i.e. without coating were stored and similarly analyzed.

2.3.8. Gasification of pellets in fixed bed reactor

Fixed bed combustion is generally used for energy production from waste biomass at various scales (Gilbert et al., 2009). In this study, the gasification of the canola meal pellets was investigated in the fixed bed reactor. The fixed bed reactor has been frequently applied for fundamental studies of biomass gasification process (Gilbert et al., 2009; Dalai et al., 2003, 2009). Details of fixed bed gasification set up are mentioned elsewhere (Tilay et al., 2014). The experiments were carried out using different gasifying agents, including steam, oxygen (O_2) and carbon dioxide (CO_2), at atmospheric pressure in a two-stage reactor system. The operating parameters were chosen based on the maximum LHV for syngas obtained by Tilay et al. (2014) for canola meal (steam/650 °C/0.2 ER; O_2/650 °C/0.4 ER and CO_2/750 °C/0.2 ER). Both reactors, first stage (10.5 mm ID × 500 mm length) and second stage (10.5 mm ID × 370 mm length) were made of Inconel tubing. Previously weighed two pellets of optimized formulation were loaded in the first stage reactor and silica sand (100–45 mesh) was used to form a 70 mm high fixed bed in the second stage reactor. The temperature of both furnaces was controlled by two temperature controllers (Eurotherm model 2132, USA). Carrier gas (Argon) was used at the flow rate of 44 ml/min. Both reactors were heated up to same final temperature with the same heating rate (25 °C/min). The injection of the gasifying agent (steam/O_2/CO_2) and simultaneous collection of gas sample was started as soon as the first reactor reaches to a temperature of 250 °C. Total run time was set at 60 min after injection of gasifying agent. The volume of gas collected was measured at 25 ± 2 °C and 1 atm in the water column over a solution of sodium chloride (17 wt%) and gas samples were collected in sampling bags for GC analysis. The reactors were cooled down with the continuous flow of argon. In case of steam gasification, a pre-calibrated syringe pump was used to inject water into the gasifier.

The product gas samples were analyzed for the permanent gases H_2, N_2, CO_2, CO, CH_4, CmHn (light hydrocarbons) using GC (Agilent Technologies, model 7890A, ON, Canada) with flame ionization detector (FID) and thermal conductivity detector (TCD). The tar sample was collected in a condenser placed in an ice bath. After cooling down the system, tubings were washed with acetone to collect residual tar. Acetone was evaporated using a rotary vacuum evaporator to collect tar free from acetone. Char sample remaining in gasifier was weighed and subjected to elemental analysis (C, H, N, S). The GC calibration was performed prior to analysis using certified standard gas procured from Praxair Products Inc, Saskatoon, SK, Canada.

2.3.9. *Scanning electron microscope (SEM)*

The optimized sample (amongst formulations 1–17) was selected for SEM analysis to study the binding characteristics of the canola meal pellets by fracture surface analysis. The fracture surface was prepared by manually snapping a pellet into two parts. Each pellet for analysis was snapped in the similar way. A tiny notch was given in the center of the pellet using a sharp blade and the pellet was snapped. The fractured surface was examined away from the notch carefully. The pellet samples were placed on carbon tapes and then coated with a thin layer of gold–carbon in an inert (argon) atmosphere using Agar Sputter Coater (Desk-1 sputter coater, Denton, USA). Electron micrographs were recorded using a SU 6600 Hitachi Field Emission SEM (Hitachi High-Technologies Europe GmbH, Krefeld, Germany) operated at 12 kV.

3. Results and discussion

3.1. *Box–Behnken design to study the effect of additives and moisture content on pellet quality*

It can be seen from Table 1 that, added binder (*A*), lubricant (*B*), and moisture content (*C*) in the formulation have shown noticeable effects on the density, durability and hardness of the produced canola meal pellet. It is a well known fact that the moisture content affects to a great extent on pelletizing properties and product quality, thus has been the subject of several studies (Larsson et al., 2013; Mahapatra et al., 2010; Serrano et al., 2011; Mani et al., 2006). These studies comprised densification of variety of biomass at different moisture content and their effects on pellet quality were analyzed. It was found that, the optimum level of moisture content varies with the different raw material. In case of canola meal, the low amount of moisture content (8 wt%) was found to be less effective to compact the canola meal particles and to produce less durable pellets with decreased relaxed density and pellet hardness (see formulations 5–6 and 9–10, Table 1). Each formulation with variable concentrations of additives and moisture content has behaved differently. In addition to the formulation composition, the inherent feed composition (various biomass constituents) also exhibits different characteristic behavior during the compression process in the presence of moisture and heat. The feed composition of canola meal used in this study contains about 12.0 wt% of lignin, 15.7 wt% of cellulose, 10.0 wt% of hemicellulose, 40.3 wt% of crude protein and about 9.5 wt% of residual canola oil (Tilay et al., 2014). In the presence of moisture, protein molecules tend to denaturate with high temperature. Denaturation of protein involves the actual breakdown of three-dimensional protein structure. Upon further cooling of pellets after densification, the protein molecules re-associate and establish the bonds with the other feed particles (Thomas et al., 1998). Along with the added binder, the protein present in the canola meal acts as an additional binding agent, building pellet more durable and better pellet hardness. According to Thomas (1998), the increased mechanical stability of proteins, increases the pellet quality characteristics which is mainly due to the covalent binding, electrostatic interactions, van der Waals forces and hydrogen bonds. These feed components such as proteins, lignin and hemicellulose of canola meal exhibit different properties in the presence of water. Thomas et al. (1997) have described that water during compression changes the structure of the surrounding particles to such an extend that makes to bind feed particles to each other. In addition, the applied heat during the process enhances the binding properties and consequently increases the pellet quality. In case of canola meal pellets, it was observed that (see formulations 8 and 11, Table 1) high moisture content (12 wt%) pellets with larger concentration of binder (3.5–5.0 wt%) produced pellets with comparatively high durability (>61%) and hardness (>63 N). This might be due to the enough amount of binding agent (protein and binder) available to bind the feed particles firmly to each other and more moisture (12 wt%) favors this bonding additionally. Cavalcanti and Behnke (2005a) reported that formulation is the single most significant variable which can affect the physical quality of the pellet. They investigated the effects of the feed composition such as proteins, fats, starch and fibers on the corn meal pellet quality using the simplex mixture design. It was observed that increased in protein concentration did lead to negative effects on pellet durability whereas an increase in starch concentration did lead to improved pellet durability. A similar experiment by Cavalcanti and Behnke (2005b) observed the effects of nutrients on soybean meal and yellow dent corn pellet durability. In this case, the highest durability was found with added protein showing a positive impact on overall pellet quality.

The reasonable quality pellets were observed with 12 wt% moisture and 5 wt% binder having a pellet durability 64% and pellet hardness 71 N (formulation 8) whereas poor quality pellets having a pellet durability 41% and pellet hardness 30 N were observed with 8 wt% moisture content and 2 wt% binder (formulation 5) in case of formulation study using BBD. The pellets extruded from the pellet mill achieve high temperature due to applied heat and the friction between the biomass and the press channel wall. This elevated temperature helps in redistribution of moisture present in the feed via evaporation and condensation. This causes an increase in the movement of water molecules among the feed particles (Thomas and van der Poel, 1996). In the case of low moisture content (8 wt%), total binding forces available are less, thus produce pellet with low quality due to more brittle and loosely bound particles. Furthermore, applied load, heat and moisture during the process facilitate the plastic deformation of the feed particles. In the case of biomass pellets, plastic deformation occurs at the glass transition temperature of the amorphous material (such as hemicellulose, starch and lignin) in the biomass (Thomas, 2010). Kaliyan and Morey (2009) reported that apart from the traditional reasoning behind requirement of moisture for the binding of feed particles with van der Waals forces and hydrogen bonds during the densification process, the glass transition temperatures of inherent polymers present in the feed are also responsible for the compressibility of the feed material. Therefore, in addition to the role of moisture, which facilities the binding between the feed particles, the physicochemical changes, i.e. thermal softening (glass transition) of the inherent canola meal biomass polymers (lignin and hemicellulose) and binder plays important role in densification. Kelley et al. (1987) investigated mechanical thermal analysis of the lignin and hemicellulose polymers in the wood cell wall. The relationship between the glass transition temperature and moisture was studied using the Kwei equation. From the study it was observed that moisture affects the glass transition temperature of the lignin and hemicellulose to a great extend. In case of canola meal, less moisture content (<8 wt%), glass transition temperature of polymers is high and thus low melting of the lignin and hemicellulose produces less durable pellets with low hardness values (see formulations 5–6 and 9–10). Some pellets have a tendency to expand after pelletization, a phenomenon called as “spring back effect” of pellet (Wolfgang et al., 2012). It signifies the binding quality between the biomass feed particles within a pellet.

In this study, the lubricant was found to give mixed effects depending on the concentration of binder and moisture content available in the formulation. The most common lubricant utilized for densification of biomass is vegetable oil to reduce the friction between the die wall and feedstock. Generally, lubricants are used by hardwood pellet manufacturers due to the fibrous nature of the feedstock (Thomas, 2010). An overall lubricant added to the canola meal formulation and inherent residual oil content show improvement in the throughput and thus assist in decreasing the energy consumption. Similar effects were observed by Cavalcanti and Behnke (2005b) with the addition of proteins in the feed formulation. However, Briggs et al. (1999) reported that the addition of oils or fat in the feed formulation will not always affect pellet energy consumption. This might be the result of an interaction with other processing parameters or due to physical properties of the fats used.

The regression prediction equation was developed for dependent parameters such as durability and hardness with independent parameters such as moisture content, binder and lubricant concentration for quality canola meal pellets. Final equation in terms of actual factors of response surface quadratic model:

$$\begin{aligned}\text{Durability}\,(\%) = {} & -171.84 + 7.44 \times A + 14.01 \times B + 38.28 \times C \\ & + 0.98 \times A^2 - 4.02 \times B^2 - 1.47 \times C^2 + 1.94 \\ & \times A \times B - 1.39 \times A \times C - 0.91 \times B \times C \end{aligned} \quad (3)$$

Pellet hardness response after square root transformation:

$$\begin{aligned}\text{Hardness}\,(\text{N}) = {} & -12.84 - 1.73 \times A + 9.21 \times B + 1.93 \times C - 0.01 \\ & \times A^2 - 0.04 \times B^2 - 0.05 \times C^2 - 0.28 \times A \times B \\ & + 0.28 \times A \times C - 0.75 \times B \times C \end{aligned} \quad (4)$$

where *A*, added binder; *B*, lubricant and *C*, moisture content.

These equations can be utilized to get the maximum value of canola meal pellet durability and hardness with R^2 value 0.997 and 0.999 respectively. The “Pred R^2” of 0.963 for durability was in reasonable agreement with the “Adj R^2” of 0.993. “Adeq Precision” value of 49.9 measures the S/N ratio. The S/N ratio greater than 4 is required. In case of durability, the S/N ratio of 49.9 indicates an adequate signal. Similarly, in case of hardness response, the “Pred R^2” of 0.988 was in reasonable agreement with the “Adj

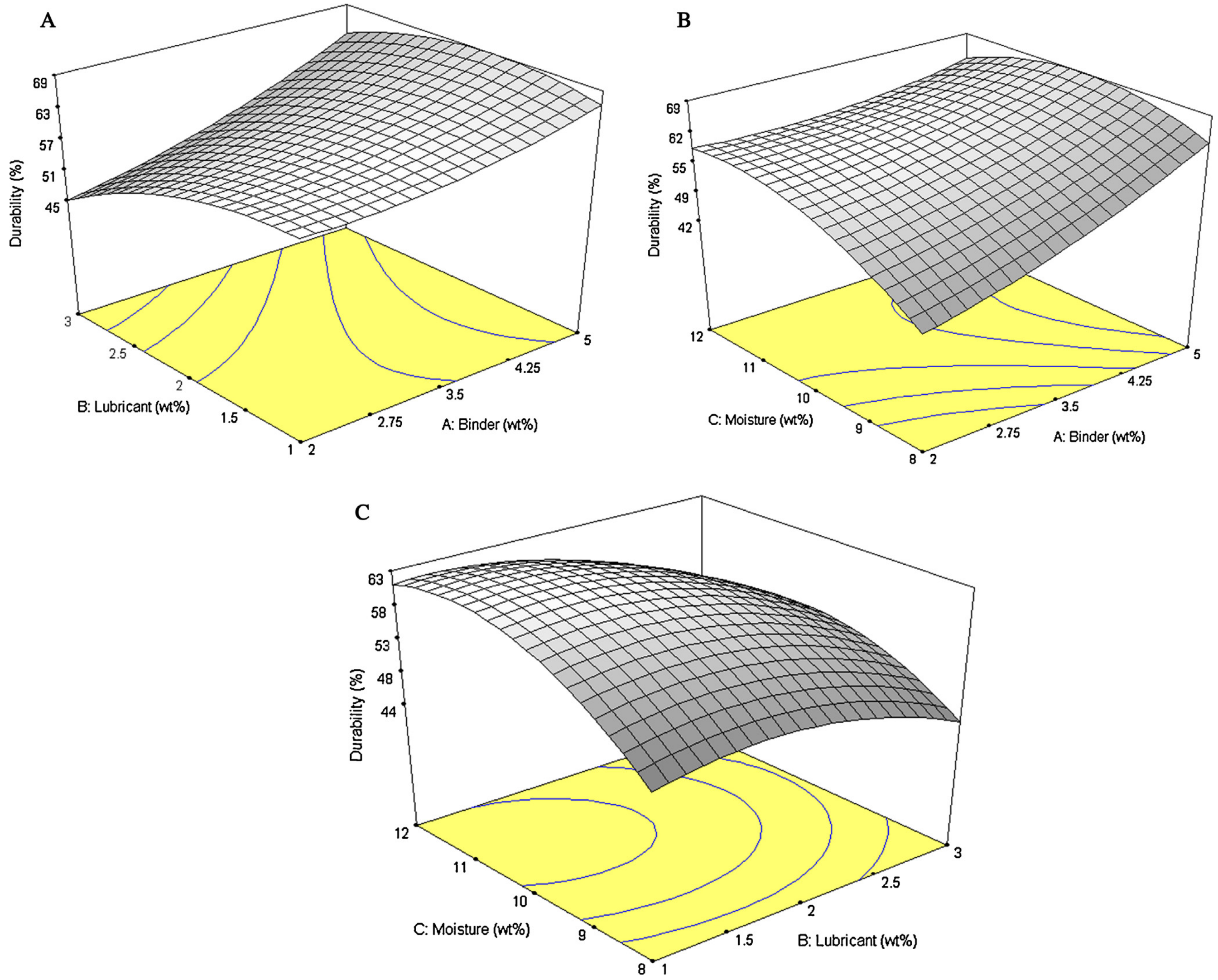

Fig. 3. Response surface interaction graphs for durability response obtained for canola meal pellet.

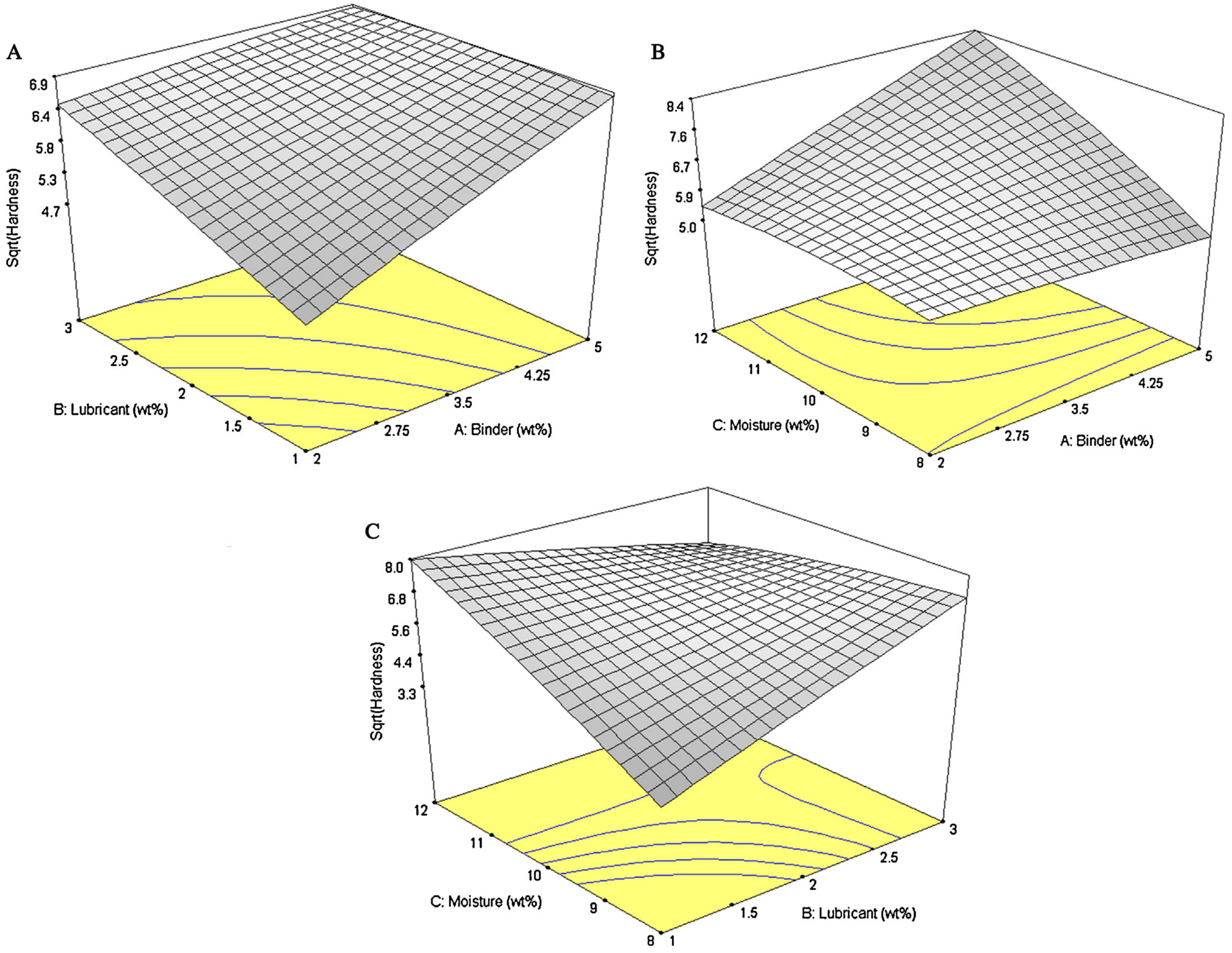

Fig. 4. Response surface interaction graphs for hardness response obtained for canola meal pellet.

R^2” of 0.997. Besides durability, “Adeq Precision” value 102.7 indicates an adequate signal. The interaction graphs of independent parameters are shown in Figs. 3 and 4. The ANOVA for the response surface quadratic model is described in Table 2. The probability values less than 0.05 indicate significant model terms. The durability and hardness model *F* values 263.7 and 610.8 indicate that the model is significant. The “Lack of Fit F-Value” 4.1 and 1.6 indicates that “Lack of Fit” is non-significant and non-significant “Lack of Fit” is desirable for the model.

3.2. Effect of physical parameters on pellet quality

It was observed that applied load and temperature were found to be the key processing parameters affecting both the compression process and pellet quality of canola meal. The formulation 8 (binder – 5 wt%; lubricant – 2 wt% and moisture – 12 wt%) obtained from BBD was considered as an optimized formulation and used further for all studies. From Table 3, it was found that with increase in applied load, pellet quality decreases. When the applied load increased to 4000 N and 4500 N, the feed material from the die

Table 2
Analysis of variance (ANOVA) obtained from BBD for durability and hardness response.

Source	Durability		Hardness		
	F value	Prob > *F*	*F* value	Prob > *F*	
Model	263.72	<0.0001	610.79	<0.0001	Significant
A (binder)	802.23	<0.0001	778.00	<0.0001	
B (lubricant)	378.45	<0.0001	367.57	<0.0001	
C (moisture content)	342.49	<0.0001	1335.20	<0.0001	
A^2	49.82	0.0002	0.98	0.3545	
B^2	163.90	<0.0001	2.44	0.1624	
C^2	351.07	<0.0001	38.01	0.0005	
AB	81.10	<0.0001	171.46	<0.0001	
AC	167.22	<0.0001	671.55	<0.0001	
BC	31.95	0.0008	2129.70	<0.0001	
Lack of fit	4.07	0.1042	1.60	0.3217	Not significant

Table 3
Effect of applied load and temperature on pellet density, pellet relaxed density, durability and hardness of canola meal pellets made in the single pelleting unit.

Temp. (°C)	Load (N)	Density (kg/m^3)	Density (kg/m^3)	Durability (%)	Hardness (N)
60	3500	1195 ± 7	1205 ± 6	67 ± 4	76 ± 2
	4000	1177 ± 19	1200 ± 10	62 ± 2	69 ± 2
	4500	1189 ± 12	1192 ± 5	54 ± 5	73 ± 9
Load (N)	**Temp. (°C)**	**Density (kg/m^3)**	**Density (kg/m^3)**	**Durability (%)**	**Hardness (N)**
3500	70	1243 ± 7	1215 ± 9	97 ± 1	111 ± 10
	80	1254 ± 10	1223 ± 7	99 ± 4	154 ± 6
	90	1260 ± 8	1247 ± 2	99 ± 0	189 ± 6

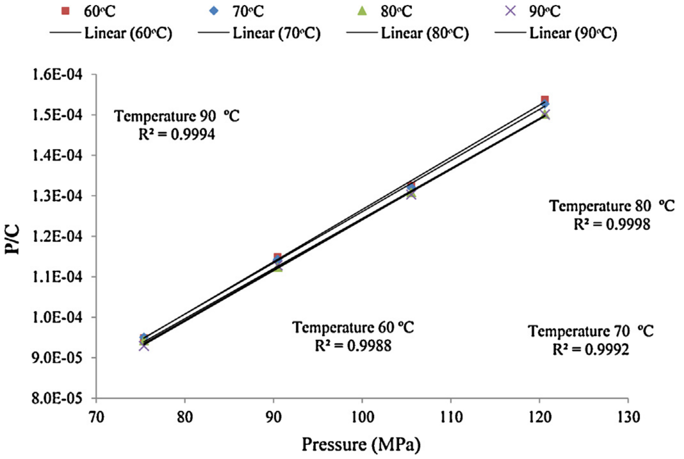

Fig. 5. Kawakita–Lüdde model experimental data for densification of canola meal.

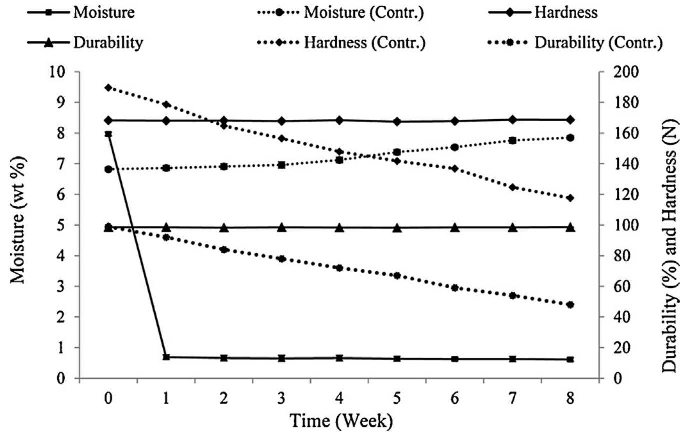

Fig. 6. Storage study of canola meal coated pellets.

wall had a tendency to come out, and failed to form a pellet. This might be due to the overload applied on feed material make it more flowable due to plastic deformation which cannot sustain at high pressure. This led to a pellet with loosely bound feed particles and thus produces less durable and low density pellet. In this case, due to poor adhesion, the pellet expands similar to a spring after compression or during storage. Hence, the optimum applied load was found to be 3500 N with high pellet density – 1205 ± 6 kg/m^3, durability – 67 ± 4% and hardness 76 ± 2 N. The pellet density of biomass compressed at different applied pressures has been studied extensively before (Adapa et al., 2009; Mani et al., 2006; Kaliyan and Morey, 2009). In all studies, it was observed that, increase in applied load or pressure, increases the pellet density; whereas in case of canola meal, it was the contradictory due to inability of feed to sustain at high applied load (>4000 N).

Pellets compressed at higher temperature (90 °C at 3500 N), expanded considerably less. This indicates that canola meal particles adhere much stronger to each other and make pellet more compact. This was reflected by a greater unit density

Table 4
Bulk density (ρ_b) and particle density (ρ_t) of canola meal; canola meal pellet density (ρ_p); compression characteristics of canola meal using Kawakita–Lüdde model.

Temp, (°C)	Pressure (MPa)	Pellet mass (g)	Pellet height (mm)	Pellet dia. (mm)	Density (ρ_p) (kg/m^3)	Kawakita compression parameters and suggested classification of feed material				
						a	b^{-1}	ab_1	R^2	Class[a]
60	75.4	0.80 ± 0.00	20.06 ± 0.10	6.41 ± 0.00	1233.6 ± 6	10^6	(−) 2	(−) 5 × 10^5	0.9988	II
	90.4	0.80 ± 0.00	20.14 ± 0.04	6.42 ± 0.03	1222.7 ± 8					
	105.5	0.80 ± 0.00	20.03 ± 0.10	6.41 ± 0.00	1231.6 ± 9					
	120.6	0.81 ± 0.00	20.21 ± 0.05	6.41 ± 0.01	1233.5 ± 9					
70	75.4	0.79 ± 0.00	20.05 ± 0.23	6.41 ± 0.01	1225.2 ± 16	10^6	(−) 0.5	(+) 2 × 10^6	0.9992	I
	90.4	0.80 ± 0.00	20.07 ± 0.06	6.42 ± 0.01	1229.7 ± 6					
	105.5	0.80 ± 0.01	19.99 ± 0.18	6.42 ± 0.01	1236.2 ± 4					
	120.6	0.80 ± 0.01	20.11 ± 0.10	6.41 ± 0.00	1233.6 ± 8					
80	75.4	0.80 ± 0.00	19.96 ± 0.19	6.41 ± 0.01	1244.4 ± 17	10^6	(+) 0.8	(+) 1.3 × 10^6	0.9998	I
	90.4	0.80 ± 0.00	19.96 ± 0.15	6.42 ± 0.02	1244.4 ± 4					
	105.5	0.80 ± 0.00	19.94 ± 0.11	6.41 ± 0.01	1253.9 ± 6					
	120.6	0.80 ± 0.00	19.94 ± 0.11	6.41 ± 0.01	1253.9 ± 6					
90	75.4	0.80 ± 0.00	19.87 ± 0.03	6.42 ± 0.00	1247.4 ± 2	10^6	(−) 0.1	(+) 1 × 10^7	0.9994	I
	90.4	0.80 ± 0.01	19.78 ± 0.28	6.41 ± 0.00	1254.9 ± 8					
	105.5	0.81 ± 0.00	19.88 ± 0.27	6.42 ± 0.00	1263.5 ± 11					
	120.6	0.79 ± 0.01	19.74 ± 0.07	6.37 ± 0.02	1243.6 ± 8					
Bulk density of canola meal (ρ_b)					737.6 ± 10					
Particle density of canola meal (ρ_t)					1387.1 ± 6					

[a] Classification based on the initial rearrangement (Nordström et al., 2009).

$1247 \pm 2\,\text{kg/m}^3$ of pellets pressed at 90 °C with durability $99 \pm 0\%$ and hardness 189 ± 6 N. Generally, pellet leaving the press channel is at a lower temperature than the actual applied heat to the die wall. This may be due to the poor heat transfer between the metal surface of the press channel and the feed (Serrano et al., 2011). Thus, in the case of applied temperature of 90 °C, the pellet leaving the press channel was around 70 °C. This temperature is sufficient for crude protein present in canola meal to denaturate and melts lignin, hemicellulose to form the highly compact canola meal pellet.

3.3. Compression model

The pressure–density data for canola meal densification process was fitted to the Kawakita and Lüdde (1971) model as described in Eq. (2). The Kawakita compression 'a' and 'b^{-1}' parameters and the derived compression index 'ab_{I}' along with pellet height, diameter, mass and densities are described in Table 4 at different temperatures and pressures used. These parameters represent the behavior of the canola meal feed particles in different stages during the compression process, and classified according to Nordström et al. (2009).

The calculated Kawakita parameter 'a' represents the maximal engineering strain 'C_∞' of the canola meal and was found to be 10^6, at all temperatures. Mathematically, the parameter 'b^{-1}' is equal to the pressure 'P' when the value of 'C' reaches one-half of the limiting value ($C = C/2$) (Klevan et al., 2010). The value of parameter 'b^{-1}' for canola meal was different at different temperatures and ranges from 0.8 to 2. Nordström et al. (2009) have previously proposed that the value of index 'ab_{I}' can be used as an indication of the occurrence of particle rearrangement during the compression process. In case of canola meal, a high 'ab_{I}' value at a temperature of 70–90 °C indicates a high degree of particle rearrangement during compression, where a material is considered by a high value of the parameter 'a' combined with a low 'b^{-1}'. Further, it was

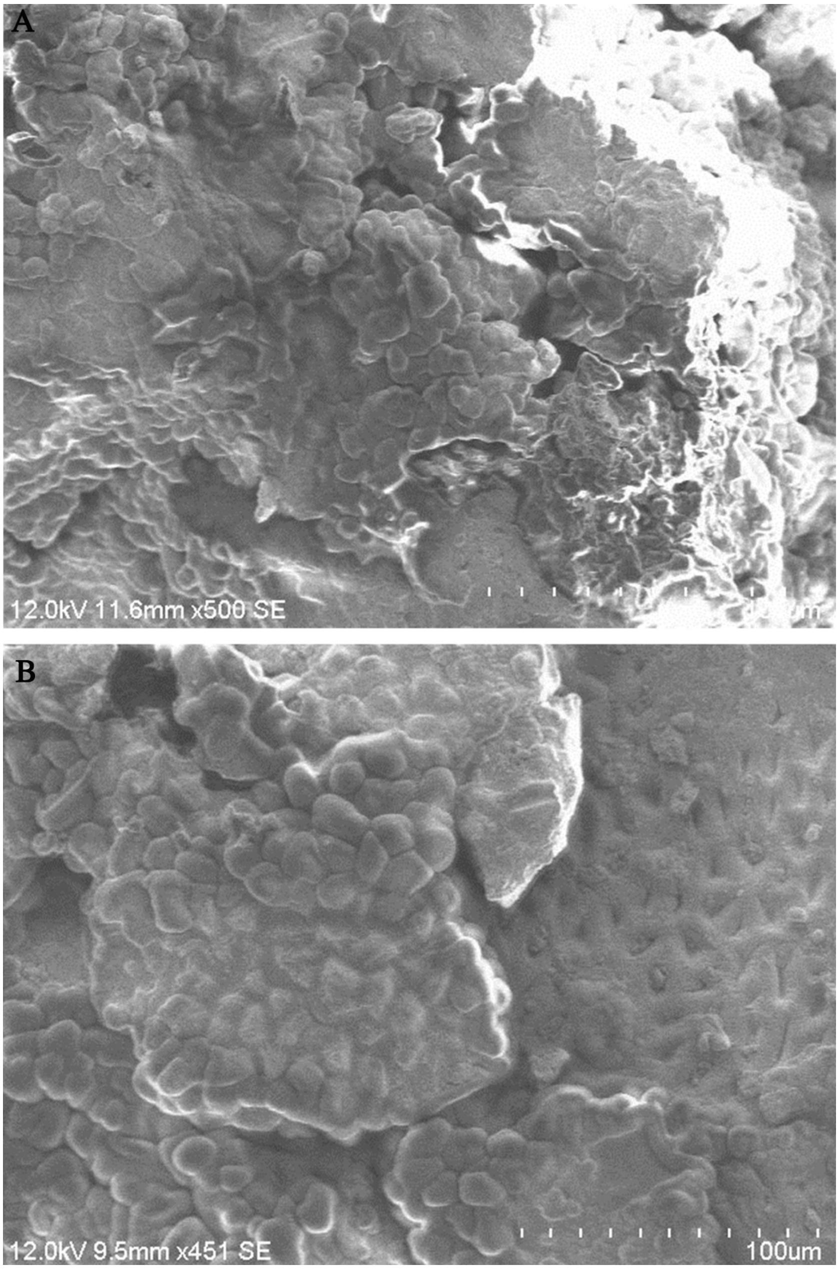

Fig. 7. SEM images of a fracture surface for canola meal pellet at different sample spots.

Table 5
Gasification of canola meal pellet using different gasifying agents.

Gasifying agent	Steam	O_2	CO_2
Equivalence ratio (ER)	0.2	0.4	0.2
Temperature (°C)	650	650	750
Gas yield (moles/kg daf-pellet)	13.7	29.8	40.0
Syngas yield (moles/kg daf-pellet)	5.8	6.4	16.6
Carbon efficiency (%)	27.4	66.5	82.7
LHV (MJ/m^3)	40.8	44.7	50.7
Total hydrocarbon (moles/kg daf-pellet)	2.7	2.8	1.9
Char (wt%)	24.9	6.8	19.8
Tar (wt%)	31.7	25.8	45.8

found that the canola meal feed shows different behavior of particle rearrangement during compression at different temperatures. The value of index 'ab_I' > 0.1 for the temperature 70–90 °C (see Table 4) falls under Class I type of powder, where particles show significant compression behavior due to primary rearrangement of the original particles followed by fragmentation and deformation dependent on their fragmentation pattern. However, very low value of index 'ab_I' $\leq$ 0.1 in case of temperature of 60 °C, falls under Class II type of powders showing limited compression due to secondary particle rearrangement and particle deformation (Nordström et al., 2009). In all cases, the value of R^2 was found to be $\geq$0.999 illustrating the perfect fit for the Kawakita and Lüdde (1971) model (Fig. 5).

3.4. *Pellet coating and storage study*

The moisture content, durability and hardness of the canola meal coated pellets against the exposure time are presented in Fig. 6. It was clearly seen that the coated canola meal pellets retain the original pellet quality characteristics up to eight weeks. The rapid drop in moisture content from 8.0 to 0.7 wt% may be due to the fact that the evaporation of residual isopropyl alcohol takes out the bound moisture from the pellet during storage, thus retaining the durability up to ~98% and hardness ~168 N. In case of control pellets (without coating), moisture content of pellets increases from 6.8 ± 0.03 to 7.9 ± 0.02 wt%, making pellets less durable. Exposure to ambient temperature has decreased the durability and hardness of controlled pellets from 99 to 48% and from 189.6 to 117.6 N, respectively. In comparison to the controlled canola meal pellets, moisture resistant canola meal pellets have obviously shown improved stability in an ambient environment. Thus, the coating of pellets made it as a one of the alternatives to make moisture resistant quality pellets with the high durability and hardness value.

3.5. *Gasification of pellets in fixed bed reactor*

The non-catalytic gasification of canola meal pellets using different gasifying agents has shown that canola meal pellets can be successfully utilized as fuel pellet for the production of biogas with high LHV. From Table 5, it can be seen that gasification of canola meal pellet using CO_2 as a gasifying agent has produced a maximum gas yield (40.0 moles/kg daf-pellet) and syngas yield (16.6 moles/kg daf-pellet) with maximum carbon conversion efficiency about 82.7%. The LHV of product gas using all three gasifying agents was in the range of 40–50 MJ/m^3. The char produced as a by-product was in the range of 19–25 wt% for steam and CO_2 gasifying agent, whereas in case of O_2, it is the lowest as O_2 is a strong gasifying agent which burns maximum amount of carbon in the feed (Tilay et al., 2014). Similarly, tar produced was found to be maximum in the case of the CO_2 gasifying agent which is due to the introduction of carbon into the system producing more tar (Tilay et al., 2014).

3.6. *Scanning electron microscope (SEM)*

The SEM images of the optimized pellet sample showed that the tight bonding occurred between different particles. Images taken at the same magnification of the same sample at different locations (Fig. 7A and B) have provided a deeper insight into the bonding mechanisms of the canola meal pellet. The proteins and biomass polymer particles stick to each other, allowing low void spaces. This showed that binder and biomass polymers (lignin and hemicelluloses) have exceeded the glass transition temperatures during the pelletization process, allowing them to form solid bridges between adjacent particles. The globular portion of the images might be due to the denaturation of globular protein particles. Denaturation at high temperature made it to adhere to the adjacent particles.

4. Conclusion

The effects of moisture content, additives, applied load and temperature on the mechanical properties of canola meal pellets were studied. It was found that all these parameters significantly affected the pellet quality. Optimized pellets with 99% durability and 189 N hardness were produced at an applied load of 3500 N and a temperature of 90 °C with the 5 wt% binder, 2 wt% lubricant and 12 wt% moisture content. A linear Kawakita and Lüdde (1971) model was developed to determine the effects of compressive pressure on the feed and thus the feed material was classified based on the Kawakita parameters. It was found that the canola meal particles undergo extensive rearrangement followed by fragmentation and deformation during the compression process. Further work was focused on the effect of coating agent (moisture resistant pellets) on pellet durability and hardness upon storage. The coated pellets exposed to the ambient temperature could sustain its durability (~98%) and hardness (~168 N) up to eight weeks without any moisture uptake, whereas control pellets (uncoated) lose its durability (~48%) and hardness (~117 N) due to moisture uptake. The SEM images of pellets exhibited effective bonding characteristics between different particles in the canola meal feed. The gasification of canola meal pellets using different gasifying agents showed that canola meal pellets can be used as a substitute for other biomass solid fuels. The product gas produced using steam, O_2 and CO_2 gasifying agent were found to have LHV in the range of 40–50 MJ/m^3. Therefore, canola meal pellets provide an alternative for biofuel production from waste biomass feedstocks in the form of moisture-resistant quality fuel pellets.

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